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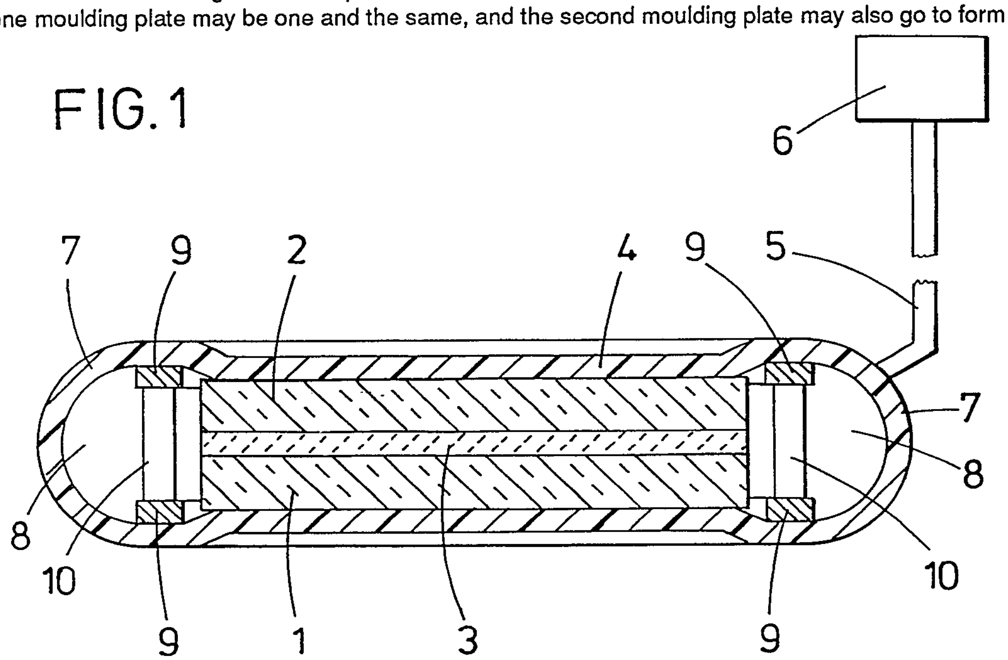
(54) Transparent fire-resistant panels

(57) Transparent, fire-resistant, glazing panels comprise at least one layer of intumescent material (3) bonded to at least one structural ply (1, 2) of the panel. Such a panel comprises a structural ply which is bonded to an intumescent layer, which has been formed by compacting grains of an intumescent hydrated metal salt, and which has a total water content in the range 20 to 26% inclusive. This promotes the retention of good optical properties despite ageing.

Such a panel may be manufactured by a method in which grains of an intumescent hydrated metal salt having a total water content of between 22% and 26% by weight are distributed as a layer on a surface of a ply to be incorporated into the panel, and in which, while the layer of grains is sandwiched between a pair of moulding plates, that layer is subjected to heat and pressure conditions to degas and compact it and cause it to become bonded to that surface of the ply of the panel. That ply and one moulding plate may be one and the same, and the second moulding plate may also go to form a ply of the panel.

FIG. 1



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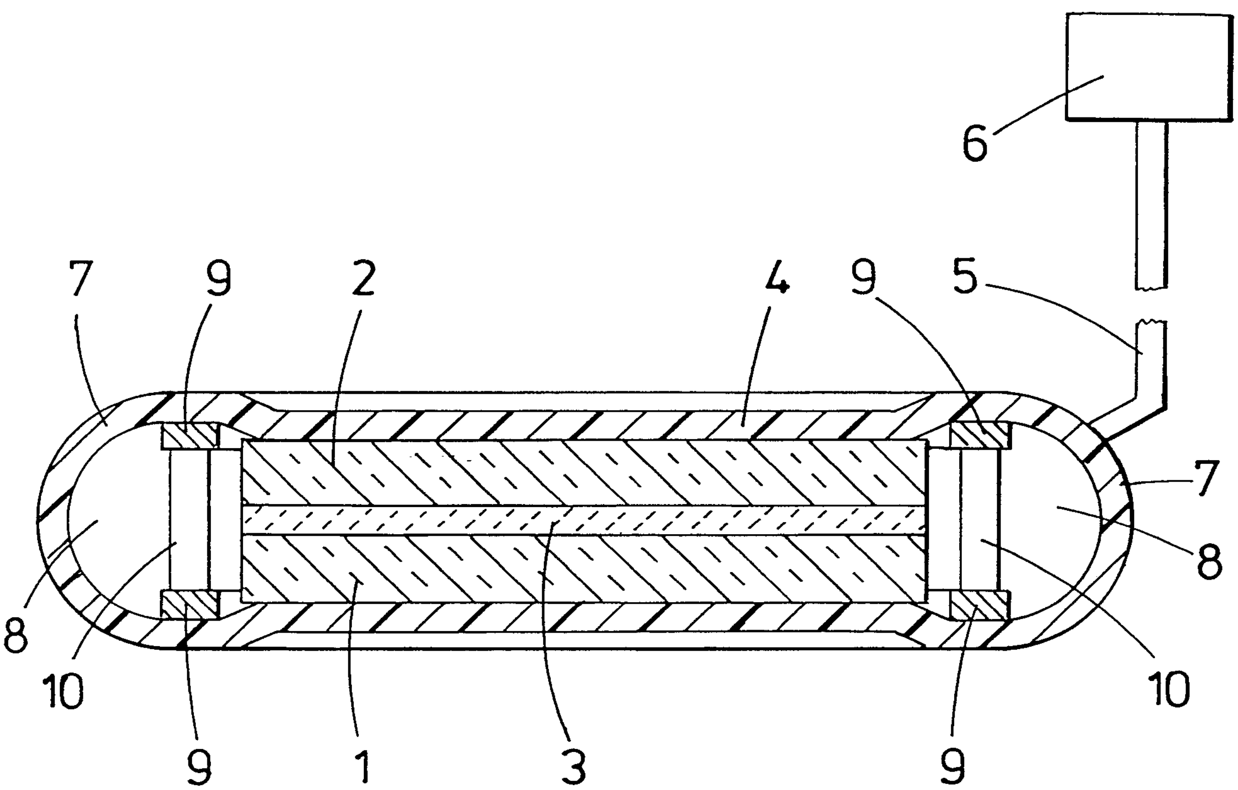

FIG. 1

TRANSPARENT FIRE-RESISTANT PANELS

This invention relates to a transparent fire-resistant glazing panel having at least one layer of intumescent material bonded to at least one structural ply of the panel. The invention includes a method of manufacturing such a panel.

Layers of intumescent material are often associated with sheets of glazing material to form fire-resistant panels. For example such a layer may be sandwiched between two glass sheets. Very important uses of such panels are as transparent screens which permit illumination of a screened area and as closures of viewing apertures of rooms or other enclosures where there may be a risk of fire.

Typically, the effectiveness of such panels is tested by mounting them in a wall of a furnace whose interior temperature is then increased according to a pre-determined schedule. Details of such a test are specified in International Standard No ISO 834-1975. The fire-resistance test procedure set out in that Standard is also referred to in International Standard ISO 9051-1990 which speaks specifically of the fire-resistance characteristics of glazed assemblies. It is appropriate to quote some passages from that latter Standard here.

> "Glass is a non-combustible material and therefore will not contribute to or propagate fire.
>
> "Glass if affected by heat may fracture by thermal shock or may soften and then not be held by the frame. Only certain types of glazed assemblies are, therefore, recognized as fire-resisting. The ability of glazed assemblies to resist fire depends on the type of glazed products, glazing method, frame type, pane size, fixing method and the type of construction surrounding the glazed area.
>
> "Some transparent and translucent glazed assemblies can meet requirements for stability and integrity (RE), and in some cases insulation (REI, where R is for "*resistance*", E for "*étanchéité*" and I for "*insulation*").
>
> "Not only is the possibility of direct fire propagation through openings caused by glass breakage to be considered for fire protection precautions: it may also be necessary to take into account the heat transmitted through the glazed assembly, which may still be intact, as such heat may cause ignition of combustible materials.
>
> "Glazed assemblies of fire-resistance according to class RE under the fire conditions as defined in ISO 834 provide, for a given time, stability and

integrity. The temperature of the unexposed side is not taken into account.

"Glazed assemblies of fire-resistance according to class REI under the fire conditions as defined in ISO 834 provide, for a given time, stability, integrity and insulation."

There are different grades of fire-resistant panel, and among those commonly recognised are grades which correspond to panels which are effective barriers against flames and fumes (i.e. Class RE) for periods of 15, 30, 45, 60, 90 and 120 minutes. Other grades correspond to panels which are effective barriers against the passage of flames and fumes and also have certain insulating properties (i.e. Class REI), again for periods of, for example 15, 30, 45, 60, 90 and 120 minutes.

The insulation properties which a panel must afford in order to meet the standard to REI level are, briefly, that no point of the surface which is exposed to the exterior of the furnace may undergo an increase in temperature of more than 180°C above its initial (ambient) temperature, and the mean temperature increase of that face must not exceed 140°C. Such panels belonging to class REI may also form barriers against the transmission of infra-red radiation from the seat of a fire.

It is extremely important that the layer of intumescent material of such a panel should have good fire-resistant properties during the course of a fire, and also that it should maintain acceptable optical properties until it starts to become tumid during the course of a fire.

Hydrated mêtal salts, for example metal silicates, especially alkali metal silicates have been used for the manufacture of such panels for some years. Typically, the layers incorporated in the finished panels have a water content of between 29% and 35%. Here and elsewhere in this specification, references to water content are references to water content as a proportion by weight of the intumescent material used to form the layer, or as a proportion by weight of the intumescent material incorporated as a layer in a finished panel (prior to the outbreak of a fire and consequent modification of that layer). During the course of a fire, the water of hydration is driven off by the heat of the fire, and the layer of intumescent material becomes converted to an opaque foam which serves as a barrier to both radiated and conducted heat, and that layer may also serve to bond together structural sheets of the panel such as sheets of glass which may become shattered by thermal shock due to the fire. The effectiveness of the panel as a

barrier against the passage of flames and fumes is thus also prolonged.

The effectiveness of a panel of hitherto known type as a fire screen depends on several factors. The effectiveness of a laminate consisting of a single layer of a given intumescent material sandwiched between two glass sheets of a given thickness increases with the thickness of the intumescent layer. For a previously known panel of given mass per unit area, that is for the same total thicknesses of glass and intumescent material, the effectiveness of such a known panel may be increased by forming it as a five-ply laminate in which there are two intumescent layers held between three sheets of glass. Thus, a laminate of three sheets of glass each 4 mm thick sandwiching two intumescent layers each 1 mm thick has often been found to be more effective than a laminate of two 6 mm glass sheets sandwiching a single intumescent layer 2 mm in thickness. A same effectiveness can therefore be achieved by using a thinner panel comprising more plies. It is clearly desirable to provide highly effective fire-resistant panels which are of low mass per unit area, but forming panels of four or more plies can be expensive.

Another problem which is associated with the use of hydrated metal salt layers as intumescent material is the ageing of the material over the course of time. This ageing is apparent as deterioration of the optical properties of the panel, for example a reduction in the transparency of the hydrated intumescent material which in turn reduces the transparency of the panel. Such a deterioration in the properties of a panel will clearly be prejudicial to its use.

The problem of deteriorating optical properties on ageing of a fire-resistant panel incorporating an intumescent layer has been known for many years, and various attempts have been made to solve this problem. A major cause of the deterioration in optical properties has been the appearance of micro-bubbles in or at the surface of the layer, and it is known to make up the layer by the *in situ* drying of a solution of the hydrated metal salt using water which has been degassed, and to take care when preparing the solution not to agitate the solution to such an extent that air or other gas becomes re-dissolved so that it can reappear as the dried layer ages. While this gives an improvement in the ageing properties of the panel, it is not entirely satisfactory if such a panel is to be used in circumstances where it is exposed to mild heat, for example due to direct sunlight. It is also known to add to the hydrated metal salt a stabilising agent such as a partially dissociated nitrogenous organic compound, for example a quaternary ammonium compound such as tetramethylammonium hydroxide, and

this indeed does give better results.

It is an object of the present invention to provide a transparent fire-resistant panel having good ageing properties which do not depend essentially on the use of such an additive, and which also offers good fire-resistance properties during the course of a fire.

According to this invention, there is provided a transparent, fire-resistant, glazing panel comprising at least one layer of intumescent material bonded to at least one structural ply of the panel, characterised in that said panel comprises a said structural ply which is bonded to a said intumescent layer, such layer having been formed by compacting grains of an intumescent hydrated metal salt and having a total water content in the range 20 to 26% inclusive.

We have found that such a panel is less susceptible to deterioration of its optical properties over the course of time than is a known panel in which the water content is somewhat higher. In fact the ageing properties of a panel according to this invention are, other things being equal, better than those of a panel whose intumescent material has a higher water content in the range 29 % to 34 % and which incorporates a stabilising agent such as tetramethylammonium hydroxide. This is rather surprising, and it is not at all clear to us why this beneficial result should accrue by the use of an intumescent layer having a lower water content.

It is also surprising that such a panel can have improved fire-resistance properties, because one would have expected that the lower water content in the intumescent layer would in fact reduce the effectiveness of the panel because there would be less foaming action during the course of a fire. In fact we have found that a three-ply laminate incorporating a single intumescent layer sandwiched between a pair of vitreous sheets in accordance with this invention tends to have a better fire resistance than a three-ply laminate of similar dimensions whose intumescent layer has a higher water content. Thus the invention affords the additional benefit of enabling the achievement of a same fire resistance by using a thinner and lighter panel without incurring the additional complications and cost of increasing the number of plies of the laminate.

Furthermore, the rather low water content of the intumescent layer, a maximum of 26%, promotes its hardness so that it is more physically stable and has less tendency to creep. Working in such a way as to produce a layer having a water content of not less than 20% is advantageous for the production of panels

having good transparency.

It is preferred that said layer has a water content not less than 22%. The presence of such proportions of water in the intumescent layer afford very good foaming properties during the course of a fire, and they also permit the formation of a hard and compact layer of intumescent material which retains good optical properties over the course of time. Optimally, said layer has a total water content of not less than 23%.

Also for the best results, it is preferred that said layer has a total water content of not more than 25%, since this promotes the retention of good optical properties despite ageing of the panel.

Such a layer of grains can readily be compacted by subjecting it to suitable temperature and pressure conditions to form a layer in which the individual grains are not visible to the naked eye so that the layer presents a uniform appearance, and is transparent. However the presence of such grains can be revealed, for example by ultra-sonic scanning or by microscopic examination, and accordingly grain boundaries, albeit invisible, are believed to remain within the layer. It is thought that this structure of the layer may have some effect on the behaviour of the intumescent material during the course of a fire, and perhaps even on the properties of the layer before such a fire. One possible contributory factor for the potential improved fire-resistance of a panel according to this invention is that though the grain boundaries disappear to the naked eye, they may remain and act as a multiplicity of sites for bubble formation on reaction of the intumescent material during the course of a fire thus resulting in a fine foamed structure which has a good and uniform insulating effect over the area of the panel.

Advantageously, said grains have a maximum dimension less than 700 μm, and they are preferably sized above 10 μm for example they may have a maximum dimension of between 150 μm and 500 μm. This promotes the ease with which the grains may be formed into a compact layer, and it may also have a beneficial effect on the behaviour of the intumescent material during the course of a fire. Panels incorporating this preferred feature of the invention have been found to give a fine and uniform foam structure when subjected to intense heat such as would be afforded by a fire. This is believed to be at least mainly due to the relatively low water content of the intumescent material as compared with that hitherto used in the manufacture of fire-resistant panels, and to the fact that there is a residual graininess in the compacted layer, but the fineness of the

residual grain structure in the layer may also be a contributory factor.

As has been stated, the efficacy of a fire-resistant panel during the course of a fire depends at least in part on the thickness of the (or each) layer of intumescent material. It is preferred that the or a said layer of intumescent material has a thickness of between 0.1 mm and 5.0 mm. Layers even as thin as 0.1 mm can give adequate short-term protection against fire, though naturally, better protection is afforded by thicker layers. In general, increasing the thickness of such a layer beyond 5 mm does not give a commensurate increase in the degree of protection afforded, and we have also found that it is also more difficult to form thicker compact layers having good optical properties.

The intumescent material may be any of a large number of hydrated metal salts, though the use of an alkali metal salt is preferred. Examples of suitable alkali metal salts which can be used in hydrated form are as follows: potassium aluminate, potassium plumbate, potassium stannate, sodium stannate, sodium aluminium sulphate, potassium aluminium sulphate, sodium borate, potassium borate, sodium orthophosphates, and potassium silicate. For reasons of cost and efficacy, however, it is preferred that said intumescent material comprises hydrated sodium silicate, which may optionally be mixed with hydrated potassium silicate.

Preferably, said panel comprises two structural plies which are laminated together via a said intumescent layer. This is a very stable and simple panel structure. In its simplest form, such a laminate could consist of two sheets of glazing material which are bonded directly to either side of an intumescent layer. Alternatively, where a higher degree of fire-protection is desired, two intumescent layers could be bonded to form a laminated panel with three structural plies of glazing material. It will readily be appreciated that if greater fire-protection is required, then two or more such panels could themselves be laminated together, for example using intervening adhesive material such as polyvinyl butyral in a process known *per se* in the laminated glazing art.

We have already mentioned that the ageing properties of a panel according to this invention are, other things being equal, better than those of a panel whose intumescent material has a higher water content in the range 29 % to 34 % and which incorporates a stabilising agent. It would be natural to think therefore that there would be no incentive for the use of such a stabilising agent in a panel whose intumescent material was of low humidity according to the invention, since such a panel already has very good ageing properties. The use of such a

stabilising agent can, however, give a further improvement in the ageing properties of a panel according to the invention, and furthermore, it can have a different and quite unexpected advantage in that the use of such an agent can promote the fire-resistance properties of the layer during the course of a fire, and this is of particular benefit in a panel having a plurality of layers of intumescent material incorporating such an additive. It is accordingly preferred that such layer of intumescent material contains at least one silicate-stabilising agent.

Preferably, the silicate-stabilising agent comprises at least one nitrogenous organic compound, e.g. an amino compound, which is at least partially dissociated, for example a quaternary ammonium compound such as tetramethylammonium hydroxide. We believe that the incorporation of a stabilising agent such as tetramethylammonium hydroxide in accordance with these preferred features of this invention not only gives an additional benefit as regards the properties of the panel on ageing, but also can have a beneficial effect on the foam produced during the course of a fire and so contribute to the fire-resistance efficacy of the panel.

A panel according to the invention can be manufactured quite simply, and the invention includes a method of manufacturing a light- transmitting, fire-resistant, glazing panel comprising at least one layer of intumescent material bonded to at least one structural ply of the panel, characterised in that grains of an intumescent hydrated metal salt having a total water content of between 22% and 26% by weight are distributed as a layer on a surface of a ply to be incorporated into the panel, and in that while the layer of grains is sandwiched between a pair of moulding plates, that layer is subjected to heat and pressure conditions to degas and compact it and cause it to become bonded to that surface of the ply of the panel.

Such a method is very simple to put into operation, and it can be performed using apparatus already known per se in the laminated glazing art.

In addition to conferring very good ageing and fire-resistance properties on the panel, the choice of intumescent grains having the specified humidity has other advantages. The use of intumescent grains having a water content not exceeding 26% promotes excellent retention of the optical properties of the panel despite its ageing, and such grains are also easy to handle prior to and during manufacture of the panel. Intumescent grains having a water content not less than 22% are quite easy to compact into hard and transparent layers, or at least into layers which will be transparent when they are bonded between a pair of

transparent sheets. This is not to say that the resulting intumescent layer will necessarily have a water content of 22% or more. Some humidity is likely to be drawn off during degassing, but the mean water content of the layer will only be very slightly less than the mean water content of the grains from which it was formed. We have found that the difference in water content between the grains and the layer is at most 2% and may be negligible, so that, for example, a layer formed from grains with a mean water content of 25% will have a mean water content of between 23% and 25%. Where grains of lower humidity are used for forming the layer it is most desirable to control the degassing conditions so that only a little water is drawn off: it is undesirable to have a mean water content in the layer which is below 20%, and it is most preferred that such water content is not below 22%.

During degassing and compaction, the intumescent layer becomes bonded to the ply of the panel with which it is in contact. That ply may be constituted by a film of thermoplastics adhesive material for subsequent bonding to structural ply of the panel such as a sheets of glass, but unless this is particularly desired for some special reason, it adds an extra step to the manufacturing process, and it is accordingly preferred that said layer of grains is distributed onto a surface of a vitreous sheet which is to become incorporated into the panel and which also constitutes a said moulding plate.

If it is desired that the other said moulding plate should not become bonded to the resulting intumescent layer, that plate may be treated in an appropriate manner, for example with a silicone, but it is preferred that the other said moulding plate is constituted by, or faced with, a ply which is to become incorporated into the panel and to which the intumescent layer becomes bonded. The intumescent layer may thus be sandwiched between two plies of the panel which is formed into a laminate at the same time as the layer is degassed and compacted. Indeed the whole panel may be assembled and formed into a laminate by that degassing and compacting treatment. The panel may then be transferred to an autoclave for a subsequent high-pressure bonding step if that is required.

It will be appreciated that any desired number of successively alternating plies of glazing material and intumescent material may be laminated together in that way, but that the difficulty of manufacturing a laminate of good optical properties increases with the number of layers of intumescent material, especially if three or more such layers are to be compacted simultaneously and if the

sandwich assembly is to be subjected to heating during compaction and/or bonding of such layers as will be adverted to below.

It will also be appreciated that two or more such panels consisting of alternate layers of glazing material and intumescent material may themselves be laminated together using adhesive thermoplastics film material if greater fire-resistance is required. Such a procedure has practical advantages where it is desired to incorporate several layers of intumescent material.

As a practical example, it might be desired to produce a fire-resistant panel having four layers of intumescent material each about 1.5 mm thick. We have found that layers of grains of intumescent material for forming such compacted layers may need to be up to about seven times as thick as the compacted layers, so in practical terms such a panel may shrink in thickness by up to about 36 mm during degassing and bonding. Manufacture is simplified by forming two panels each having two such intumescent layers and laminating those panels together using a thermoplastics adhesive film material such as polyvinyl butyral. The presence of such a thermoplastics adhesive film material can also have a beneficial effect on the fire-resistance properties of the panel by limiting the propagation of cracks due to thermal shock.

In the most preferred embodiments of the invention, said grains have a total water content of not less than 23%, and preferably, such water content is not more than 25% by weight. The presence of such proportions of water in the intumescent grains affords the resulting layer very good foaming properties during the course of a fire, and also permits the formation of a hard and compact layer of intumescent material which possesses and retains good optical properties over the course of time.

Preferably, said grains are sized so that at least 90% by weight of them have a maximum dimension of less than 700 μm, and preferably in the range 10 μm to 500 μm. Grains of such sizes are convenient to handle, and they impart to the resulting compacted layer a structure which is believed to be beneficial for affording good fire-resistance properties as has been stated. Such grain sizes are also especially convenient for forming into layers of the thicknesses most especially in view, for example layers formed to a thickness between 0.1 mm and 5.0 mm.

Advantageously, for at least part of the time during degassing and bonding, the layer of intumescent material is subjected to a temperature of at least 80°C. Heating the intumescent material to such a temperature promotes

degassing and compaction, and also bonding to a ply of the panel. It will be appreciated that the intumescent material must not be subjected to such high temperatures as would, having regard to the pressure applied to the material, give rise to premature foaming of the intumescent material. It may be noted here that it is very much easier to ensure that a single intumescent layer, or each of two intumescent layers, of a panel is subjected to the optimum heating schedule than it is to ensure that each of three or more layers is optimally heated, if only because the central layer(s) will be more shielded from the heat source by other plies of the panel than are the outer layers.

Advantageously, during degassing and bonding, the layer of intumescent material is subjected to a pressure of less than 30 kPa. This allows excellent degassing of the intumescent material.

We have previously referred to the use of additives in an intumescent layer for improving its ageing properties. The use of such an additive can have other unexpected advantages in promoting fire-resistance during the course of a fire as has also been referred to. Advantageously, therefore, such intumescent material contains at least one silicate-stabilising agent, and preferably the silicate-stabilising agent comprises at least one nitrogenous organic compound, e.g. an amino compound, which is at least partially dissociated, for example a quaternary ammonium compound such as tetramethylammonium hydroxide.

Preferred embodiments of the invention will now be described with reference to the accompanying drawings in which :

Figure 1 is a schematic view of apparatus for degassing and compacting a layer of intumescent material in a method according to the invention.

In Figure 1, a panel sandwich has been made up to consist of two sheets of glass 1, 2 and an inter-layer 3 composed of grains of intumescent material having a total water content of between 22% and 26%. The grains are of a size fraction which passes a screen having mesh openings of 500 μm but which is retained by a screen having mesh openings of 150 μm. The grains are simply spread loosely on a first sheet of glass and levelled off with a granular layer thickness of seven times the desired final thickness of the compacted layer to be formed. The panel sandwich is enclosed within an envelope 4. The envelope is connected by a vacuum line 5 to a pump 6 by which sub-atmospheric pressure can be maintained within the envelope to keep the space between the sheets 1, 2 subjected to suction. When the pump is operated, the top and bottom walls of the envelope are drawn against the main external faces of the enclosed sandwich, and the glass

sheets 1, 2 act as moulding plates for compacting the granular intumescent layer 3. The envelope at least at its peripheral zone 7, is sufficiently rigid to resist collapse against the edges of the sandwich so that a space 8 at a sub-atmospheric pressure maintained by the pump 6 is preserved within the envelope 4, around the edges of the sandwich 1, 2, 3.

The use of an envelope which encloses the sandwich affords the advantage that the size of the envelope in relation to the dimension of the sandwich is not critical. The envelope can be easily applied to sandwich of a range of different sizes. Furthermore, the use of such an envelope facilitates the application of uniform pressure over the whole area of the main faces of the sandwich during its treatment, so that reaction forces arising from pressure differences between the environment in which the envelope is placed and the space within the envelope will not be such as to cause flexure of the outer sheets 1, 2 of the sandwich. Such flexure could lead to the formation of bubbles in the margins of the layer 3 and can also lead to a non-flat final product.

In a variant of the apparatus just described, optional bracing means are provided for supporting reaction forces arising from pressure differences between the interior and exterior of the envelope 4. In Figure 1, such bracing means are shown as a pair of frames 9 of the same shape as but slightly larger than the sandwich assembly 1, 2, 3 which are held spaced apart by a plurality of pillars such as 10. The frames 9 are so spaced by the pillars 10 as to hold the envelope slightly away from the edges of the assembly when the intumescent layer has shrunk in thickness to its final compacted dimension shown in the drawing.

Heaters (not shown) may be disposed over the upper and lower faces of the envelope 4 for heating the sandwiched intumescent material 3 to assist in compaction and bonding of the sandwich.

A sandwich assembly may be treated by the aspirating means shown in Figure 1 in a simple process in which the exterior of the envelope 4 is always subjected to atmospheric pressure. In an example of the process, the pump 6 is switched on to reduce the pressure within the envelope, that is the pressure acting on the edges of the assembly in the edge space 8, to a value below 30 kPa. The precise optimum value will depend upon the water content of the intumescent grains used. The required value may be reached after some few minutes, and it is maintained for a further 100 minutes. The sandwich assembly is initially at room temperature (20°C). The sandwich assembly in the envelope 4 is heated so that it reaches a temperature of 90°C after 45 minutes.

After the required degassing, the pressure in the envelope is allowed to return to atmospheric over a period of about 15 minutes. At the end of this time, the granular layer 3 is found to have become compacted to such an extent that boundaries between the grains have become invisible to the naked eye, and the sandwich assembly is found to be bonded together as a transparent laminated panel. Of course this panel may then be transferred to an autoclave for a subsequent high-pressure bonding step if desired.

Water loss from the intumescent material due to aspiration during its compaction into the layer is found to be less than 2% in relation to the weight of the layer.

EXAMPLE 1

A series of panels was made by the process described above using glass sheets each 3 mm thick with an intervening intumescent layer of hydrated sodium silicate 1.5 mm thick, having a total water content of between 23.5% and 24.5%. The layer of each such panel was formed from grains having a total water content of 24.5% which had been screened so that their dimension was between 150 μm and 500 μm. The weight ratio of SiO_2 to Na_2O in the sodium silicate was between 3.3 and 3.4 to 1. The intumescent grains did not include any tetramethylammonium hydroxide as fire-resistance promoting additive.

A series of similarly sized comparative test panels was also made by a classical process in which, to form each such panel, hydrated sodium silicate solution was dried in situ on a glass sheet 3 mm thick to form layers averaging 1.8 mm in thickness (range 1.5 mm to 2.1 mm) with a total water content of between 29 % and 34 %. The solution included 0.25 % by weight of tetramethylammonium hydroxide as anti-ageing additive. The weight ratio of SiO_2 to Na_2O in the sodium silicate was again between 3.3 and 3.4 to 1. A second sheet of 3 mm thick glass was bonded to that layer to form a laminated panel.

The panels were framed in substantially identical frames to form glazed assemblies for testing by the procedure set forth in International Standard No ISO 834-1975.

Two glazed assemblies, one from each series of panels, were then mounted side by side in the wall of a furnace, and the furnace was heated according to the required predetermined schedule in order to test the stability and integrity of the two assemblies as barriers against the passage of flames and fumes according to Class RE. It was found that the comparative test assembly

satisfied ISO 834 to RE level for 30 minutes but not for 45 minutes. The assembly comprising a panel according to the invention satisfied ISO 834 to RE level for more than 60 minutes.

The two types of panel were also subjected to ageing tests. In a first test, panels were maintained at 80°C for fifteen days. At the end of that period, no micro-bubbles were apparent in a panel according to the invention, whereas a considerable number of bubbles was apparent in a comparative test panel, so that it exhibited haze despite the presence of the silicate-stabilising agent in its intumescent layer. Haze was only apparent in the panel according to the invention after 30 days. In a second test, panels were subjected to U.V. irradiation for 500 hours. The panel according to the invention exhibited no micro- bubbles after that time, but the comparative test panel showed more than twice as many micro-bubbles as it did after the first ageing test.

EXAMPLE 2

Two further series of panels according to the invention were made using the same starting materials as given in Example 1. In these series, the panels consisted of three glass sheets each of 3 mm thickness, and two intumescent inter-layers each 1.5 mm in thickness. In one such series of panels according to the invention, the intumescent layers included a proportion of tetramethylammonium hydroxide; in the other such series, no such additive was present. The tetramethylammonium hydroxide was incorporated by adding it to the silicate solution from which the grains were formed in a proportion of 0.125% by weight. A series of comparative test panels of the same structure was made from the solution of hydrated sodium silicate with added tetramethylammonium hydroxide as was specified for the comparative test panels of Example 1. The intumescent layers of such comparative test panels again had an average thickness of 1.8 mm.

The panels were again framed in substantially identical frames to form glazed assemblies for testing by the procedure set forth in International Standard No ISO 834-1975.

Such glazing assemblies were then mounted side by side in the wall of a furnace, and the furnace was heated according to the required predetermined schedule in order to test the stability, integrity and insulation afforded by the two series of assemblies according to Class REI. It was found that the various assemblies were all able to maintain their integrity as a barrier against the passage of flames and fumes and to satisfy the insulation requirements of Class

REI for between 30 and 35 minutes.

Other members of each series of glazing assembly were subjected to the ageing tests referred to in Example 1. It was found that according to each test, all the panels according to the invention gave better results than the comparative test panels, and also that of the panels according to the invention, those which had intumescent material incorporating tetramethylammonium hydroxide gave better results than those which did not.

EXAMPLE 3

Two further series of panels according to the invention were made as specified in Example 2 save that one outer glass sheet of each panel was 2 mm thick rather than 3 mm. The panels of each series were laminated together with their 2 mm glass sheets to the inside using intervening films of polyvinyl butyral (PVB) 0.76 mm in thickness. Thus, in one series of such PVB-laminated panels, each incorporated four 1.5 mm layers of hydrated sodium silicate with tetramethylammonium hydroxide, while in the other series, no tetramethylammonium hydroxide was present.

The panels were again framed in substantially identical frames to form glazed assemblies for testing by the procedure set forth in International Standard No ISO 834-1975.

The assemblies were then mounted side by side in the wall of a furnace, and the furnace was heated according to the required predetermined schedule in order to test the efficacy of the two series of panels, again according to Class REI. It was found that those assemblies whose panels did not incorporate tetramethylammonium hydroxide were able to maintain their integrity as a barrier against the passage of flames and fumes and to satisfy the insulation requirements of Class REI for periods of 55 to 70 minutes. The assemblies according to the invention whose panels did incorporate tetramethylammonium hydroxide remained effective as flame- and fume- proof barriers and satisfied the insulation requirements of Class REI for periods of 70 to 80 minutes.

EXAMPLE 4

A further series of glazed assemblies was made by laminating together and then framing three panels each themselves according to this invention and formed as specified in Example 2, using intervening layers of polyvinyl butyral 0.76 mm in thickness. The assemblies were then mounted side by side in the wall of a furnace, and the furnace was heated according to the required predetermined schedule in order to test their efficacy, again according to Class

REI. It was found that these assemblies according to the invention remained effective as a flame- and fume- proof barrier and satisfied the insulation requirements of Class REI for more than 90 minutes, and when tetramethylammonium hydroxide was present as fire-resistance promoting additive, they satisfied the requirements of Class REI for up to 110 minutes.

EXAMPLE 5

Two fire-resistant panels according to the invention were manufactured each to contain three sheets of glass 3 mm in thickness and two intumescent layers each 0.6 mm in thickness. In one panel, the intumescent material included tetramethylammonium hydroxide as fire-resistance promoting additive as specified in Example 2: in the other panel, no such additive was used.

Framed assemblies incorporating the two panels were then tested as to their stability, integrity and insulation (Class REI) on exposure to fire. The panel without the additive failed at 34 minutes. The panel with the additive resisted the effects of the test for between 35 and 36 minutes.

EXAMPLE 6

Two fire-resistant panels were manufactured each to contain three sheets of glass respectively 3 mm, 8 mm and 3 mm in thickness and two intumescent layers. In one panel, each intumescent layer was formed in accordance with the invention as specified in Example 1, to a thickness of 2.5 mm: in the other panel, the intumescent layers were formed to thicknesses of about 1.8 mm by the classical technique as specified in relation to the comparative test panel mentioned in Example 1.

Framed assemblies incorporating the two panels were then tested as to their stability, integrity and insulation (Class REI) on exposure to fire. The comparative test panel failed at 40 minutes. The panel according to the invention resisted the effects of the test for 50 minutes.

CLAIMS

1. A transparent, fire-resistant, glazing panel comprising at least one layer of intumescent material bonded to at least one structural ply of the panel, characterised in that said panel comprises a said structural ply which is bonded to a said intumescent layer, such layer having been formed by compacting grains of an intumescent hydrated metal salt, and having a total water content in the range 20 to 26% inclusive.

2. A panel according to claim 1, wherein said panel comprises two structural plies which are laminated together via a said intumescent layer.

3. A panel according to claim 1 or 2, wherein said layer has a total water content not less than 22%.

4. A panel according to claim 3, wherein said layer has a total water content not less than 23%.

5. A panel according to any preceding claim, wherein said layer has a total water content not more than 25%.

6. A panel according to any preceding claim, wherein said grains have a maximum dimension less than 700 μm, and they are preferably sized between 150 μm and 500 μm.

7. A panel according to any preceding claim, wherein the or a said layer of intumescent material has a thickness of between 0.1 mm and 5.0 mm.

8. A panel according to any preceding claim, wherein said intumescent material comprises hydrated sodium silicate.

9. A panel according to any preceding claim, wherein such layer of intumescent material contains at least one silicate-stabilising agent.

10. A panel according to claim 9, wherein the silicate-stabilising agent comprises at least one nitrogenous organic compound, e.g. an amino compound, which is at least partially dissociated, for example a quaternary ammonium compound such as tetramethylammonium hydroxide.

11. A method of manufacturing a transparent, fire-resistant, glazing panel comprising at least one layer of intumescent material bonded to at least one structural ply of the panel, characterised in that grains of an intumescent hydrated metal salt having a total water content of between 22% and 26% by weight are distributed as a layer on a surface of a ply to be incorporated into the panel, and in that while the layer of grains is sandwiched between a pair of moulding plates, that layer is subjected to heat and pressure conditions to degas and compact it and cause it to become bonded to that surface of the ply of the panel.

12. A method according to claim 11, wherein said layer of grains is distributed onto a surface of a vitreous sheet which is to become incorporated into the panel and which also constitutes a said moulding plate.

13. A method according to claim 12, wherein the other said moulding plate is constituted by, or faced with, a ply which is to become incorporated into the panel and to which the intumescent layer becomes bonded.

14. A method according to any of claims 11 to 13, wherein said grains have a total water content not less than 23% by weight.

15. A method according to any of claims 11 to 14, wherein said grains have a total water content not more than 25%.

16. A method according to any of claims 11 to 15, wherein said grains are sized so that at least 90% by weight of them have a maximum dimension of less than 700 μm, and preferably in the range 150 μm to 500 μm.

17. A method according to any of claims 11 to 16, wherein said layer is formed to a thickness between 0.1 mm and 5.0 mm.

18. A method according to any of claims 11 to 17, wherein said layer comprises hydrated sodium silicate.

19. A method according to any of claims 11 to 18, wherein, for at least part of the time during degassing and bonding, the layer of intumescent material is subjected to a temperature of at least 80°C.

20. A method according to any of claims 11 to 19, wherein, during degassing and bonding, the layer of intumescent material is subjected to a pressure of less than 30 kPa.

21. A method according to any of claims 11 to 20, wherein such intumescent material contains at least one silicate-stabilising agent.

22. A method according to claim 21, wherein the silicate-stabilising agent comprises at least one nitrogenous organic compound, e.g. an amino compound, which is at least partially dissociated, for example a quaternary ammonium compound such as tetramethylammonium hydroxide.

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Databases (see over)

(i) UK Patent Office

(ii) ONLINE DATABASES: WPI, CLAIMS

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Documents considered relevant following a search in respect of claims 1 TO 22

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
X	GB 2199535 A (GLAVERBEL) eg page 4 lines 29 to 30	1-5 and 7-10
X	GB 2155852 A (GLAVERBEL) eg page 1 lines 102 to 116	1-8 and 11-20
X	GB 2023452 A (BFG GLASSGROUP) eg page 2 lines 37 to 47	1-8 and 11-20
X	GB 1590837 A (BFG GLASSGROUP) eg page 5 lines 14 to 18	1-8 and 11-20
X	GB 1290699 A (BADISCHE) eg page 1 lines 51 to 55	1-8 and 11-20

Category	Identity of document and relevant passages	Rele .t to claim(s)

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